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NO DRAWINGS.

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COMPLETE SPECIFICATION.

Improvements in or relating to Detergent Compositions.

We, THOMAS HEDLEY & CO. LIMITED, a British Company, of Phoenix Buildings, Collingwood Street, Newcastle-upon-Tyne, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement :—

This invention comprises improvements in or relating to detergent compositions, and is more particularly concerned with such compositions containing organic soapless detergents which can be produced in the form of bars or tablets.

Conventional toilet soap has a major disadvantage in use in the tendency towards formation of insoluble calcium and other insoluble soaps when the soap is used in hard water. These insoluble soaps appear as an unpleasant curd or scum in the water, and are frequently left as an unsightly ring of scum around the basin or bath used.

Many organic soapless detergents have a considerable advantage over soap in that they do not give rise to insoluble products when used in hard water. However, for a variety of reasons it has proved extremely difficult to provide organic soapless detergents in the form of a bar or tablet which is satisfactory for toilet purposes.

A satisfactory detergent bar for toilet use should desirably have the following properties :—

It should provide a good lather quickly, and should have good cleansing properties on the skin in hard or soft water, hand-hot or cold ;

It should be mild to the skin and not produce irritation even after repeated use ;

In use it should have a soap-like feel rather than a sticky, smeary or greasy feel ;

It should not form curd or scum in hard water ;

It should not tend to crack when the tablet is allowed to dry out after use, and it should not become slushy or smeary if left standing in a soap dish containing a layer of water ;

It should not have any unpleasant intrinsic odour or colour ;

It should be capable of manufacture into bar or tablet form, by conventional soap making equipment such as mills, plodders and stamps.

The present invention is based on the discovery that a detergent composition which has all the above requirements in a high degree can be produced by selection of certain ingredients within certain ranges of proportions.

According to the invention, a detergent composition, which can be produced in the form of bars or tablets, comprises by weight of the total composition (a) from 7% to 65% of a mixture of organic soapless detergents containing (1) a water-soluble salt of an N-fatty acyl taurine and/or a water-soluble salt of an N-fatty acyl-N-methyl taurine in amount by weight 40% to 80% of the mixture, the fatty acyl group in each case having from 10 to 20 carbon atoms, and (2) a water-soluble salt of a fatty acyl isethionic acid in which the fatty acyl group has from 10 to 20 carbon atoms, and/or a water-soluble salt of an alkyl sulphuric acid in which the alkyl group has from 10 to 20 carbon atoms, in amount by weight 60% to 20% of the mixture ; (b) from 0 to 25% of fatty acid having from 12 to 20 carbon atoms, and having an iodine value not exceeding 10 ; (c) from 2 to 15% of moisture ; (d) up to 30% by weight of the total organic soapless detergent (a) of inorganic salt ; and (e) at least 25% of alkali metal soap or mixture of alkali

metal and calcium, magnesium or zinc soaps derived from C_9 to C_{25} fatty acids, at least 50% of which is C_{12}—C_{18} fatty acids, the weight ratio of calcium, magnesium or zinc soap to alkali metal soap not exceeding unity.

Other normal additives for such products, such as perfume, colour, preservative, opacifier and deodorant, may also be included in the compositions.

Water-soluble salts of N-fatty acyl taurine, or N-fatty acyl-N-methyl taurine for use according to the invention have the general formula :—

$$R.CO—N(R^1)—CH_2.CH_2.SO_3M$$

where R.CO is an acyl radical derived from a fatty acid, including a mixture of fatty acids, having from 10 to 20 carbon atoms in the molecule, R^1 is hydrogen or a methyl group, and M is a cationic atom or radical forming a water-soluble salt with the taurine derivative. Preferably the acyl radical RCO is derived from a fatty acid mixture which occurs naturally in animal or vegetable oils, particularly suitable compounds being obtained when RCO is derived from coconut fatty acids. M is preferably an alkali metal, though it may also be other metals or radicals giving a water-soluble salt, such as ammonium or substituted ammonium. Particularly suitable compounds are the sodium and potassium salts.

Suitable water-soluble salts of fatty acyl isethionic acids which are suitable for the purposes of the invention have the general formula :—

$$R^{11}.CO.O.CH_2.CH_2.SO_3M^{11}$$

where $R^{11}.CO$ and M^{11} have the same significance as RCO and M above, respectively. The particularly preferred isethionates are the sodium and potassium salts of fatty acyl isethionic acids in which the fatty acyl group is derived from a fatty acid mixture which occurs naturally in animal or vegetable oils and especially from coconut fatty acids.

The alkyl sulphates suitable for use according to the invention have the general formula :

$$R^{111}.O.SO_3.M^{111}$$

where R^{111} is an alkyl group of 10 to 20 carbon atoms and M^{111} has the same significance as M above. Particularly suitable compounds are the sodium and potassium alkyl sulphates in which the alkyl radical is derived from a fatty acid mixture which occurs naturally in animal or vegetable oils and especially from the fatty alcohols obtained by the reduction of coconut oil fatty acids.

The detergent taurides, isethionates and alkyl sulphates as obtained commercially usually contain a proportion of inorganic salts, generally sulphates and/or chlorides which are formed as by-products during the manufacture of the soapless detergent. For the purpose of the present invention it is not necessary to carry out any purification process to remove these inorganic salts unless the total of inorganic salts associated with the soapless detergent is more than 30% calculated on the weight of soapless detergent. If the inorganic salt content of the soapless detergent is higher than this limit, then the excess of inorganic salt can be removed by any of the methods known in the art, such as solvent extraction.

When included, the fatty acid constituent of the detergent composition of the invention is a fatty acid, including a mixture of fatty acids, having from 12 to 20 carbon atoms in the molecule, and having an iodine value not exceeding 10. Suitable fatty acid mixtures are those obtained from vegetable and animal fats, hardened by hydrogenation, if necessary, to meet the limit which is set on the iodine value. Fatty acids from hardened tallow are particularly suitable. Individual fatty acids such as stearic acid are also suitable.

As has already been stated, the soaps which may be included in the composition of the invention are alkali metal soaps, or mixtures of alkali metal soaps and calcium, magnesium or zinc soaps, derived from fatty acids having from 9 to 25 carbon atoms in the molecule, at least 50% of the fatty acid in the soap having from 12 to 18 carbon atoms in the molecule. The preferred soaps are those obtained from natural fats and fat mixtures such as, for example, mixtures of coconut or palm kernel oil with tallow or palm oil.

The compositions of the invention may be prepared by mixing the ingredients together in a conventional soap amalgamator, and then homogenising the mixture by passing one or more times through a toilet soap mill. The milled product may then be extruded into bar form by means of a plodder, and the bar cut into tablets and stamped.

Alternatively, the ingredients may be mixed together in the form of a slurry containing a higher proportion of water than is required in the finished product, the mixing being conveniently carried out in a soap crutcher, following which the mixed slurry is then dried to the required moisture content, for example by spray-drying or roll-drying, and the dried product milled, plodded and stamped in the normal way.

Each of the ingredients of the composition has its own function, and the properties of the finished product may be controlled by varying the proportions of the ingredients within the limits set out above. Thus the N-fatty-acyl tauride and N-fatty acyl N-methyl tauride impart good cleaning proper-

ties and hard-water scum dispersing properties, but do not lather well. The isethionate and alkyl sulphate improve the lather properties, but do not have very good cleansing or scum-dispersing properties. The fatty acid improves the feel of the product in use, and also serves as a buffering agent for the washing solution, and as a binding agent for the dry composition. The inclusion of fatty acid is unnecessary if the plasticity of the other ingredients is sufficient to enable a satisfactory bar to be produced. The alkali metal soap serves as binding agent, and as cleaning agent, and the calcium, magnesium or zinc soap serves as binding agent, and also as a lubricant in the milling and plodding operations.

The following examples are given for the purpose of illustrating the invention; all parts and percentages are by weight:—

EXAMPLE I.

		Parts.
(a) (1)	Sodium coconut tauride ..	30.70
(a) (2)	Sodium coconut isethionate	10.50
(a) (2)	Potassium alkyl (coconut) sulphate	5.35
(b)	Hardened tallow fatty acids (iodine value 3.4) ..	15.43
(d)	Inorganic salts (sodium and potassium sulphates and chlorides associated with the synthetic detergents)	9.42
(e)	Sodium soap (80% tallow, 20% coconut oil) ..	28.60

The above ingredients were mixed at 190° F. in the proportions specified, and water was added so that the slurry was readily mixed in a crutcher (about 40% moisture in the slurry).

The mixture was spray-dried to give a powder containing 3% water. This powder was mixed in a conventional toilet soap amalgamator with colour and perfume, milled by two passes over a 3-roll mill, plodded to spaghetti, and finally plodded to a bar which was cut and stamped in the normal way.

The product was a firm bar of good appearance, with very little tendency to crack or smear. In use it has good lathering and cleansing power and a soap-like feel.

EXAMPLE II.

		Parts.
(a) (1)	Sodium coconut tauride ..	8.50
(a) (2)	Sodium coconut isethionate	7.45
(a) (2)	Potassium alkyl (coconut) sulphate	1.24
(b)	Hardened tallow fatty acids (iodine value 3.4)	5.73
(d)	Inorganic salts (sodium and potassium sulphates and chlorides associated with the synthetic detergents)	4.49
(e)	Sodium soap (25% tallow, 75% coconut)	72.59

The above ingredients were mixed at 190° F. with water to give about 40% moisture in the mix, spray-dried, and then processed as described in Example I to give a bar containing approximately 3% water content.

EXAMPLE III.

		Parts.
(a) (1)	Sodium coconut tauride ..	13
(a) (2)	Potassium alkyl sulphate	10
	Palm kernel monoethanolamide	5
(e)	Zinc soap (80% tallow, 20% coconut)	20
(e)	Sodium soap (80% tallow, 20% coconut)	30
(d)	Inorganic salts (sodium and potassium sulphates and chlorides associated with the synthetic detergent)	12
(c)	Water	10

The above ingredients were intimately mixed in a sigma blade mixer, excess water being added to facilitate mixing. The mixture was dried to a water content of 10 parts and was then milled twice on a 3-roll mill, plodded to spaghetti and finally plodded to a bar which was cut and stamped in the normal way.

The product was a good bar with good lathering properties and a soap-like feel. The zinc soap may be replaced by magnesium soap or calcium soap with comparable results.

It is to be noted that the palm kernel monoethanolamide included in the above ingredients served as a lather stabilizer.

EXAMPLE IV.

		Parts.
(a) (1)	Sodium coconut tauride ..	14
(a) (1)	Potassium alkyl sulphate ..	9
(e)	Magnesium soap (80% tallow, 20% coconut) ..	17
(e)	Sodium soap (80% tallow, 20% coconut)	37
(d)	Inorganic salts (sodium and potassium sulphates and chlorides associated with the synthetic detergents	12
(c)	Water	10

The bars were produced in the same way as for Example III.

WHAT WE CLAIM IS:—

1. A detergent composition in bar or tablet form, or adapted to be produced into such form, which comprises by weight of the total composition (a) from 7% to 65% of a mixture of organic soapless detergents containing (1) a water-soluble salt of an N-fatty acyl taurine and/or a water-soluble salt of an N-fatty acyl-N-methyl taurine in amount

by weight 40% to 80% of the mixture, the fatty acyl group in each case having from 10 to 20 carbon atoms; and (2) a water-soluble salt of a fatty acyl isethionic acid in which the fatty acyl group has from 10 to 20 carbon atoms, and/or a water-soluble salt of an alkyl sulphuric acid in which the alkyl group has from 10 to 20 carbon atoms, in amount by weight 60% to 20% of the mixture; (b) from 0 to 25% of fatty acid having from 12 to 20 carbon atoms, and having an iodine value not exceeding 10; (c) from 2 to 15% of moisture; (d) up to 30% by weight of the total organic soapless detergent (a) of inorganic salt; and (e) at least 25% of alkali metal soap or mixture of alkali metal and calcium, magnesium or zinc soaps derived from C_9 to C_{25} fatty acids, at least 50% of which is C_{12}—C_{18} fatty acids, the weight ratio of calcium, magnesium or zinc soap to alkali metal soap not exceeding unity.

2. A detergent composition according to Claim 1 in which each of the organic soapless detergents is an alkali metal salt.

3. A detergent composition according to Claim 2 in which the alkali metal is sodium and/or potassium.

4. A detergent composition according to any of the preceding claims in which said fatty acyl group is in each case derived from a fatty acid mixture which occurs naturally in animal or vegetable oils.

5. A detergent composition according to any of the preceding claims in which said fatty acyl group is in each case derived from coconut oil fatty acids.

6. A detergent composition according to any of the preceding claims in which the alkyl radical of said alkyl sulphuric acid is derived from coconut oil fatty alcohols.

7. A detergent composition substantially as described in any of the foregoing examples.

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PROVISIONAL SPECIFICATION.

Improvements in or relating to Detergent Compositions.

We, THOMAS HEDLEY & Co. LIMITED, a British Company, of Phoenix Buildings, Collingwood Street, Newcastle-upon-Tyne, do hereby declare this invention to be described in the following statement:—

This invention comprises improvements in or relating to detergent compositions, and is more particularly concerned with such compositions containing organic soapless detergents which can be produced in the form of bars or tablets.

Conventional toilet soap has a major disadvantage in use in the tendency towards formation of insoluble calcium and other insoluble soaps when the soap is used in hard water. These insoluble soaps appear as an unpleasant curd or scum in the water, and are frequently left as an unsightly ring of scum around the basin or bath used.

Many organic soapless detergents have a considerable advantage over soap in that they do not give rise to insoluble products when used in hard water. However, for a variety of reasons it has proved extremely difficult to provide organic soapless detergents in the form of a bar or tablet which is satisfactory for toilet purposes.

A satisfactory detergent bar for toilet use should desirably have the following properties:—

It should provide a good lather quickly, and should have good cleansing properties on the skin in hard or soft water, hand-hot or cold;

It should be mild to the skin and not produce irritation even after repeated use;

In use it should have a soap-like feel rather than a sticky, smeary or greasy feel;

It should not form curd or scum in hard water;

It should not tend to crack when the tablet is allowed to dry out after use, and it should not become slushy or smeary if left standing in a soap dish containing a layer of water;

It should not have any unpleasant intrinsic odour or colour;

It should be capable of manufacture into bar or tablet form, by conventional soap making equipment such as mills, plodders and stamps.

The present invention is based on the discovery that a detergent composition which has all the above requirements in a high degree can be produced by selection of certain ingredients within certain ranges of proportions.

According to the invention, a detergent composition, which can be produced in the form of bars or tablets, comprises by weight of the total composition (a) from 7% to 65% of a mixture of organic soapless detergents containing:— (1) a water-soluble salt of an N-fatty acyl taurine and/or a water-soluble salt of an N-fatty acyl-N-methyl taurine in amount by weight 40% to 80%, the fatty acyl group in each case having from 10 to 20 carbon atoms, and (2) a water-soluble salt of

a fatty acyl isethionic acid in which the fatty acyl group has from 10 to 20 carbon atoms, and/or a water-soluble salt of an alkyl sulphuric acid in which the alkyl group has from 10 to 20 carbon atoms, in amount by weight 60% to 20%; (b) from 0 to 25% of fatty acid having from 12 to 20 carbon atoms, and having an iodine value not exceeding 10; (c) from 0 to 15% of moisture; (d) up to 30% by weight of the total organic soapless detergent (a) of inorganic salt; and (e) the balance of the composition being made up substantially of alkali metal soap or mixed alkali metal-alkaline earth metal soaps derived from C_9 to C_{25} fatty acids, at least 50% of which is C_{12}—C_{18} fatty acids, the weight ratio of alkaline earth metal soap to alkali metal soap not exceeding unity.

Other normal additives for such products, such as perfume, colour, preservative, opacifier and deodorant, may also be included in the compositions.

Water-soluble salts of N-fatty acyl taurine, or N-fatty acyl-N-methyl taurine for use according to the invention have the general formula :—

$$R.CO—N(R^1)—CH_2.CH_2.SO_3M$$

where R.CO is an acyl radical derived from a fatty acid, including a mixture of fatty acids, having from 10 to 20 carbon atoms in the molecule, R^1 is hydrogen or a methyl group, and M is a cationic atom or radical forming a water-soluble salt with the taurine derivative. Preferably the acyl radical RCO is derived from a fatty acid mixture which occurs naturally in animal and vegetable oils, particularly suitable compounds being obtained when RCO is derived from coconut fatty acids. M is preferably an alkali metal, though it may also be other metals or radicals giving a water-soluble salt, such as ammonium or substituted ammonium. Particularly suitable compounds are the sodium and potassium salts.

Suitable water-soluble salts of fatty acyl isethionic acids which are suitable for the purposes of the invention have the general formula :—

$$R_1.CO—O.CH_2.CH_2.SO_3M_1$$

where R_1CO and M_1 have the same significance as the RCO and M above, respectively. The particularly preferred isethionates are the sodium and potassium salts of fatty acyl isethionic acids in which the fatty acyl group is derived from coconut fatty acids.

The alkyl sulphates suitable for use according to the invention have the general formula :—

$$R^1_2O.SO_3M_2$$

where R^1_2 is an alkyl group of 10 to 20 carbon atoms, and M_2 has the same significance as M above. Particularly suitable compounds are the sodium and potassium alkyl sulphates in which the alkyl radical is derived from the fatty alcohols obtained by the reduction of coconut oil.

The detergent taurides, isethionates and alkyl sulphates as obtained commercially usually contain a proportion of inorganic salts, generally sulphates and/or chlorides which are formed as by-products during the manufacture of the soapless detergent. For the purpose of the present invention it is not necessary to carry out any purification process to remove these inorgainc salts unless the total of inorganic salts associated with the soapless detergent is more than 30% calculated on the weight of soapless detergent. If the inorganic salt content of the soapless detergent is higher than this limit, then the excess of inorganic salt can be removed by any of the methods known in the art, such as solvent extraction.

The fatty acid constituent of the detergent composition of the invention is a fatty acid, including a mixture of fatty acids, having from 12 to 20 carbon atoms in the molecule, and having an iodine value not exceeding 10. Suitable fatty acid mixtures are those obtained from vegetable and animal fats, hardened by hydrogenation, if necessary, to meet the limit which is set on the iodine value. Fatty acids from hardened tallow are particularly suitable.

As has already been stated, the soaps which may be included in the composition of the invention are alkali metal soaps, or mixtures of alkali metal and alkaline earth metal soaps, derived from fatty acids having from 9 to 25 carbon atoms in the molecule, at least 50% of the fatty acid in the soap having from 12 to 18 carbon atoms in the molecule. The term " alkaline earth metal soap " as used in this Specification is to be understood as including magnesium soaps in addition to calcium soaps. The preferred soaps are those obtained from natural fats and fat mixtures such as, for example, mixtures of coconut or palm kernel oil with tallow or palm oil.

The compositions of the invention may be prepared by mixing the ingredients together in a conventional soap amalgamator, and then homogenising the mixture by passing one or more times through a toilet soap mill. The milled product may then be extruded into bar form by means of a plodder, and the bar cut into tablets and stamped.

Alternatively, the ingredients may be mixed together in the form of a slurry containing a higher proportion of water than is required in the finished product, the mixing being conveniently carried out in a soap crutcher, following which the mixed slurry is then dried to the required moisture content,

for example by spray-drying or roll-drying, and the dried product milled, plodded and stamped in the normal way.

Each of the ingredients of the composition has its own function, and the properties of the finished product may be controlled by varying the proportions of the ingredients within the limits set out above. Thus the N-fatty-acyl tauride and N-fatty acyl-N-methyl tauride impart good cleaning properties and hard-water scum dispersing properties, but do not lather well. The isethionate and alkyl sulphate improve the lather properties, but do not have very good cleansing or scum-dispersing properties. The fatty acid improves the feel of the product in use, and also serves as a buffering agent for the washing solution, and as a binding agent for the dry composition. The alkali metal soap serves as binding agent, and as cleaning agent, and the alkaline earth metal soap serves as binding agent, and also as a lubricant in the milling and plodding operations.

The following examples are given for the purpose of illustrating the invention; all parts are by weight.

EXAMPLE I.

		Parts.
(a) (1)	Sodium coconut tauride ..	30.70
(a) (2)	Sodium coconut isethionate	10.50
(a) (2)	Potassium alkyl (coconut) sulphate	5.35
(b)	Hardened tallow fatty acids	15.43
(d)	Inorganic salts	9.42
(e)	Sodium soap (80% tallow, 20% coconut oil) ..	28.60

The above ingredients were mixed at 190° F. in the proportions specified, and water was added so that the slurry was readily mixed in a crutcher (about 40% moisture in the slurry).

The mixture was spray-dried to give a powder containing 3% water. This powder was mixed in a conventional toilet soap amalgamator with colour and perfume, milled by two passes over a 3-roll mill, plodded to spaghetti, and finally plodded to a bar which was cut and stamped in the normal way.

The product was a firm bar of good appearance, with very little tendency to crack or smear. In use it has good lathering and cleansing power and a soap-like feel.

EXAMPLE II.

		Parts.
(a) (1)	Sodium coconut tauride ..	8.50
(a) (2)	Sodium coconut isethionate	7.45
(a) (2)	Potassium alkyl (coconut) sulphate	1.24
(b)	Hardened tallow fatty acids	5.73
(d)	Inorganic salts	4.49
(e)	Sodium soap (25% tallow, 75% coconut)	72.59

The above ingredients were mixed at 190° F. with water to give about 40% moisture in the mix, spray-dried, and then processed as described in Example I to give a bar containing approximately 3% water content.

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